APPENDICES

APPENDIX 27

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STANDARD OPERATING PROCEDURE

TITLE: IGNITABILITY OF SOLIDS FOR WASTE CHARACTERIZATION EPA SW-846 CHAPTER 7, SECTION 7.1

(SUPERSEDES: NONE)

Prepared by: _____ 1/3/97
David Dawes and Richard Burrows

Reviewed by: _____ 1/3/97
Director, Technology

Approved by: _____
Director, Quality Assurance

Approved by: _____
Director, Environmental Health and Safety

Approved by: _____
Management

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of ignitability of solids as it pertains to waste characterization outlined in Chapter 7 of EPA SW846. It is used to identify wastes that are fire hazards under routine storage conditions or which are capable of "severely exacerbating" a fire that is already burning.

1.2. There is no instrument or method detection limit applicable to this test. Results are reported qualitatively as either "Yes" or "No" depending on the ignitability of the sample.

1.3. Applicable Matrices: Solids, including domestic and industrial wastes, sludges, and petroleum wastes.

1.4. The analytical time depends on the matrix and method of analysis. For solids, the analytical time is approximately 5 minutes.

2. SUMMARY OF METHOD

2.1. Solids are analyzed by exposure to open flame for a set length of time after which the flame is removed and the sample observed to determine its ignitability characteristic. If the material will burn, a subsequent set of tests determine ignitability according to the definitions in Chapter 7 of SW-846.

3. DEFINITIONS

3.1. Ignitability: The capability of a solid sample, under standard conditions of temperature and pressure, to cause fire through friction, moisture absorption, or spontaneous chemical changes and, when ignited, to burn so vigorously and persistently as to create a hazard.

4. INTERFERENCES

4.1. Improper storage of samples may cause loss of volatiles and lead to erroneous results.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. Because of the unknown reactivity of the materials being tested, the operator will wear a face shield in addition to safety glasses. As an alternative, the sash of the hood will be pulled down to shield the operators face.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. In order to stabilize conditions during the actual test, the fume hood when the test is being conducted may be turned off.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor and/or the EH&S Coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Ceramic crucibles, nominal 50 mL capacity.

6.2. Source of open flame: "barbecue" butane lighter, or equivalent. NOTE: a butane cigarette lighter, short wooden matches, or a high temperature propane torch are not acceptable alternatives.

7. REAGENTS AND STANDARDS

7.1. There are no reagents and standards required for this method.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are to be collected in glass bottles or jars with a minimum of headspace and refrigerated to 4 ± 2°C.

8.2. There is no specified holding time for ignitability .

9. QUALITY CONTROL

9.1. The Quanterra QA Management Plan document provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.

9.2. Sample/Sample Duplicate (SA/DU) - One Sample/Sample Duplicate pair must be processed for each QC batch per matrix, or every 20 client samples, whichever is more frequent.

9.2.1. The results of the SA/DU pair are used to determine analytical variability.

9.2.2. The SA/DU pair is evaluated qualitatively. If both the sample and its duplicate are found to be either both ignitable (YES/YES) or both not ignitable (NO/NO), the QC is considered in control.

9.2.3. The SA/DU QC is out of control if the two results are different (YES/NO). In this case, the data must be qualified appropriately (e.g. sample heterogeneity) and an explanation provided in the report narrative.

9.2.4. There are no other QC analyses applicable to this test.

10. CALIBRATION AND STANDARDIZATION

10.1. This method has no calibration or standardization requirements.

11. PROCEDURE

11.1. Any significant variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. In a fume hood, place a small quantity of sample (approx 1 gram) into the ceramic crucible. It is not necessary to weigh the sample, but a small quantity should be used to minimize any potential hazards.

11.3. Wear a face shield in addition to safety glasses, or pull the hood sash down to act as a shield. Turn off the hood (if necessary to maintain a stable flame), touch the ignition source (see section 6.2) to the sample. Keep the flame in contact with the sample for 5 ± 2 seconds.

11.4. Remove the ignition source and observe the sample.

11.5. Turn the fume hood back on. (if previously turned off).

11.6. Observations can be reduced to three broad categories:

Reaction of Sample	Classification
Sample does **not** ignite	Not ignitable, no further testing necessary
Sample ignites but burns only in contact with flame	Not ignitable, no further testing necessary
Sample ignites and burns continuously	Proceed to section 11.6.1

11.6.1. Wear a face shield in addition to safety glasses, or pull the hood sash down to act as a shield for the following steps. Place approximately 5g of solid into a crucible and stir for 10 seconds. If the sample ignites and burns continuously, then it is classified as ignitable. If not, proceed to section 11.6.2

11.6.2. Add 5 mL of water to the sample in the crucible. If the sample ignites and burns continuously, then it is classified as ignitable. If not, proceed to section 11.6.3.

11.6.3. Add approximately 5g of solid to a crucible. Heat the crucible from underneath with a burner for 30 seconds. If the sample ignites and burns continuously, then it is classified as ignitable. If not, the sample is classified as not ignitable.

12. DATA ANALYSIS AND REPORTING

12.1. If the result is "Ignitable", report as "Yes".

12.2. Samples which do not meet the "Ignitability" criteria should be reported as "No."

13. METHOD PERFORMANCE

13.1. Training Qualifications: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. Sample volumes have been reduced (maintaining proper ratios) in order to minimize laboratory waste.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The facility Environmental Health and Safety Coordinator should be contacted if additional information is required.

15.2. Dispose of samples in accordance with Quanterra waste disposal policies.

16. REFERENCES

16.1. Source Methods

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA SW846, 3rd edition, Chapter 7, "Ignitability", revision 2, September 1994.

APPENDIX 28

Section 4: 1613 Data Analysis & Reporting

Paradigm Analytical Labs - Standard Operating Procedure

Last Revised By:	Asst. Lab Director:	QA Officer:
W.M. Larkins	C.K. Cornwell	Greg Dickinson

Purpose

To describe the processes used in operating the HRGC/HRMS system, as well as the procedures followed in the generation, interpretation and review of laboratory data for Method 1613.

Summary

This SOP details how to analyze and report samples by EPA Method 1613. HRGC/HRMS is used to detect and quantitate PCDD/Fs. Samples arrive at the MS lab having been extracted and fractionated using procedures in Section 3. Analyses are grouped into 12-hour sequences that include analyses of samples and standards mixtures. Upon completion of the sequence, the analyst reviews the data associated with both standards and samples in order to confirm the validity of the sequence and to determine any potential need for re-analysis or re-extraction. The analyst generates quantitation reports and chromatograms using sophisticated software. These reports are used to generate forms that summarize the results of the analysis.

4.1 Operation of HRGC/HRMS

4.1.1 Equipment

- HP6890 GC, Micromass Autospec Ultima high resolution mass spectrometer, vortex mixer, 10-100 uL pipette

4.1.2 Procedure

- Recall the GC temperature/pressure/flow program.
- Recall the MS experiment (see Table 1).
- Perform any necessary maintenance.
- Tune the MS resolution to 100 ppm at 5% height.
- Acquire location data to calibrate the MS and print a copy of function one MS resolution.
- Inject the window defining/GC resolution/continuing calibration mix (RETCON). Evaluate descriptor-switching times for accuracy. If any window defining peaks have shifted outside the descriptor windows, adjust the switching times before injecting any samples. This injection is also used to verify that there is less than or equal to 25% peak to valley for the two close eluters of 2,3,7,8-TCDD. Print a copy of the GC resolution check. If the valleys are within specifications, proceed to calibrate or verify a previous calibration. If not, further investigation and/or maintenance may be required. Re-inject this solution after maintenance to check for improvement.
- Now that the GC/MS resolution and descriptor switching times have been verified, a series of five initial calibration standards may be injected and reviewed for method requirements. If an initial calibration already exists, a RETCON may be analyzed to verify continuing calibration. If the curve or the RETCON passes method requirements, sample analysis may begin.
- Reconstitution of a sample is accomplished by adding nonane containing the injection standards, capping the vial, and mixing well with a vortex mixer.
- Samples are injected under conditions identical to those used to establish calibration.
- A “back-end” print out of the MS resolution must be performed.
- The calibration data from a sequence is filed in a folder cabinet under the day it was analyzed and includes the all GC/MS resolution checks, window verification, valley verification, front end Retcons, run logs and window defining mix (WDM) retention time sheets.
- Each sample hardcopy should include the quant report, totals pages, deviations, chromatograms, and report forms.
- Columns: DB-225, 30 m, id 0.25 mm, 0.25 μm; DB-5MS, 60 m, id 0.25 mm, 0.25 μm.

Table 1: Mass Descriptors used for Selected Ion Recording HRMS

Function (#)	Channel (#)	Mass (amu)	Dwell Time (ms)	I.C. Delay (ms)
1	1	303.9016	100	20
1	2	305.8987	100	10
1	3	315.9419	40	10
1	4	316.9824	20	10
1	5	316.9824	(Lock)	50
1	6	317.9389	40	10
1	7	319.8965	100	10
1	8	321.8936	100	10
1	9	327.8847	40	10
1	10	331.9368	40	10
1	11	333.9339	40	10
1	12	375.8364	30	20
2	1	339.8597	100	20
2	2	341.8568	100	10
2	3	351.9000	40	10
2	4	353.8970	40	10
2	5	355.8546	100	10
2	6	357.8517	100	10
2	7	366.9792	20	10
2	8	366.9792	(Lock)	50
2	9	367.8949	40	10
2	10	369.8919	40	10
2	11	409.7974	30	20
3	1	373.8207	100	20
3	2	375.8178	100	10
3	3	380.9760	20	10
3	4	380.9760	(Lock)	50
3	5	383.8639	40	10
3	6	385.8610	40	10
3	7	389.8156	100	10
3	8	391.8127	100	10
3	9	401.8559	40	10
3	10	403.8530	40	10
3	11	445.7555	30	20
4	1	407.7818	100	20
4	2	409.7788	100	10
4	3	417.8253	40	10
4	4	419.8220	40	10
4	5	423.7767	100	10
4	6	425.7737	100	10
4	7	430.9728	20	10
4	8	430.9728	(Lock)	50
4	9	435.8169	40	10
4	10	437.8140	40	10
4	11	479.7165	30	20
5	1	441.7427	100	20
5	2	443.7398	100	10
5	3	454.9728	20	10
5	4	454.9728	(Lock)	50
5	5	457.7377	100	10
5	6	459.7348	100	10
5	7	469.7780	40	10
5	8	471.7750	40	10
5	9	513.6775	30	20

4.2 Data Generation, Interpretation and Review

Paradigm Analytical Labs defines a batch of samples as no more than 20 samples processed within a 12-hour shift. One LMB and one OPR are processed per analytical batch, following the same procedures as the field samples. Generally, soil is replaced by salt (Na_2SO_4), effluent by deionized water and biological tissues by vegetable oil. An invalid LMB or OPR requires a re-extraction of the affected samples.

4.2.1 Quality Assurance/Quality control

On an annual schedule, the laboratory shall perform Method Detection Limit studies (MDLs) for each matrix analyzed. Additionally, the laboratory shall perform and MDL study for each extraction method utilized per matrix. All MDL studies will be conducted following the guidelines set forth in 40 CFR, Part 136, appendix B and must be lower than one-third the regulatory compliance level or one third the Minimum Levels (ML) set forth in Table 2 of the reference method.

4.2.2 Initial Calibrations

The percent relative standard deviations for the mean response factors from the seventeen unlabeled standards must not exceed +/- 20%. The percent relative standard deviations from the labeled standards (i. e. extraction standards, cleanup standards and sampling standards) must not exceed +/- 35%. The signal to noise ratio for all signals present must be ≥ 10. The ion abundance ratios must be within specified control limits (see Table 2). Paradigm uses the concentrations in Table 3 to construct the initial calibration.

Table 2. Theoretical Ion Abundance Ratios and Their Control Limits

Level of Chlorination	Theoretical Ratio	Control Limits	
		Lower	Upper
4	0.77	0.65	0.89
5	1.55	1.32	1.78
6	1.24	1.05	1.43
6[a]	0.51	0.43	0.59
7	1.04	0.88	1.20
7[b]	0.44	0.37	0.51
8	0.89	0.76	1.02

[a] Used only for ^{13}C-HxCDF
[b] Used only for ^{13}C-HpCDF

A new initial calibration is required when the continuing calibration criteria below are not met. Routine maintenance may be performed to correct any failures. Any major maintenance to the analytical system such as slit cleaning, analyzer lens cleaning, magnet shifts, and detector disk changes warrant a new ICAL. At a minimum, a new initial calibration must be performed annually.

Table 3. Initial Calibration Concentrations

Analyte	CS-1	CS-2	CS-3	CS-4	CS-5
	Concentration (pg/µL)				
Unlabeled					
2378-TCDD	0.25	2	10	40	200
2378-TCDF	0.25	2	10	40	200
12378-PeCDD	1.25	10	50	200	1000
12378-PeCDF	1.25	10	50	200	1000
23478-PeCDF	1.25	10	50	200	1000
123478-HxCDD	1.25	10	50	200	1000
123678-HxCDD	1.25	10	50	200	1000
123789-HxCDD	1.25	10	50	200	1000
123478-HxCDF	1.25	10	50	200	1000
123678-HxCDF	1.25	10	50	200	1000
123789-HxCDF	1.25	10	50	200	1000
234678-HxCDF	1.25	10	50	200	1000
1234678-HpCDD	1.25	10	50	200	1000
1234678-HpCDF	1.25	10	50	200	1000
1234789-HpCDF	1.25	10	50	200	1000
OCDD	2.5	20	100	400	2000
OCDF	2.5	20	100	400	2000
Extraction Standards					
^{13}C-2378-TCDD	100	100	100	100	100
^{13}C-2378-TCDF	100	100	100	100	100
^{13}C-12378-PeCDD	100	100	100	100	100
^{13}C-12378-PeCDF	100	100	100	100	100
^{13}C-23478-PeCDF	100	100	100	100	100
^{13}C-123678-HxCDD	100	100	100	100	100
^{13}C-123478-HxCDD	100	100	100	100	100
^{13}C-123478-HxCDF	100	100	100	100	100
^{13}C-123478-HxCDF	100	100	100	100	100
^{13}C-1234678-HpCDD	100	100	100	100	100
^{13}C-1234678-HpCDF	100	100	100	100	100
^{13}C-1234789-HpCDF	100	100	100	100	100
^{13}C-OCDD	200	200	200	200	200
Cleanup Standards					
^{37}Cl-2378-TCDD	0.25	2	10	40	200
Injection Standards					
^{13}C-1234-TCDD	100	100	100	100	100
^{13}C-123789-HxCDD	100	100	100	100	100

4.2.3 Continuing Calibrations

Check that all paperwork is present. A CCal package should contain the documentation listed below.

- Pass: Run log. HRMS Resolution Checks. WDM retention time sheet. WDM chromatograms. GC performance for 2,3,7,8-TCDD. CCal quantitation page. CCal chromatograms. Injection preparation log.
- Fail: The analyst listed on the run log can provide any missing paperwork.

Review the Run log.

- Pass: Check that the 12 hour windows have not been exceeded between the front end Ccal and the last sample of the sequence.
- Fail: Re-analysis of affected samples.

Review the HRMS Resolution checks.

- Pass: Verify 100ppm width at 5% height for PFK mass 318 or higher. Compare the resolution check times to those on the run log to be sure they bracket each sequence.
- Fail: Back end resolution checks do not have to meet the front end requirements. Should one fail, an assessment should be made to determine any data quality impact.

Review the Window Defining Mix and GC Performance Documentation.

- Pass: Check that the sample numbers on the WDM sheets match those on the run log. Check that the retention times are correct for the WDM chromatograms.
- Check that the valley between 2,3,7,8-TCDD and its close eluters does not exceed 25%.
- Fail: Any missing peaks in the window-defining sample should be re-identified with a survey scan. Determine proper switching times. These must be entered into the HRMS ion function descriptors before analysis may resume. If the GC performance valley is greater than 25% instrument maintenance may be required. When a valley fails all samples must be reinjected.

Review the CCal Quantitation and Chromatograms.

- Pass: Check that all ion ratios are in specification. Verify that all compounds are within the concentration limits set by the method (see Table 4) for all front end CCals
- Fail: Routine instrument maintenance such as installing new injection port hardware, inner source cleaning, retuning, column clipping etc. will usually correct a calibration failure. If these measures do not work, a new ICal is needed.

Compound Name	CCAL (pg/µL)	Limits (pg/µL)	Compound Name	CCAL (pg/µL)	Limits (pg/µL)
2,3,7,8-TCDD	10	7.8 - 12.9	$^{13}C_{12}$-2,3,7,8-TCDD	100	82 - 121
1,2,3,7,8-PeCDD	50	39 - 65	$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	62 - 160
1,2,3,4,7,8-HxCDD	50	39 - 64	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	85 - 117
1,2,3,6,7,8-HxCDD	50	39 - 64	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	85 - 118
1,2,3,7,8,9-HxCDD	50	41 - 61	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	72 - 138
1,2,3,4,6,7,8-HpCDD	50	43 - 58	$^{13}C_{12}$-OCDD	200	96 - 415
OCDD	100	79 - 126	$^{13}C_{12}$-2,3,7,8-TCDF	100	71 - 140
2,3,7,8-TCDF	10	8.4 - 12	$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	76 - 130
1,2,3,7,8-PeCDF	50	41 - 60	$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	77 - 130
2,3,4,7,8-PeCDF	50	41 - 61	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	76 - 131
1,2,3,4,7,8-HxCDF	50	45 - 56	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	70 - 143
1,2,3,6,7,8-HxCDF	50	44 - 57	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	74 - 135
2,3,4,6,7,8-HxCDF	50	45 - 56	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	73 - 137
1,2,3,7,8,9-HxCDF	50	44 - 57	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	78 - 129
1,2,3,4,6,7,8-HpCDF	50	45 - 55	$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	77 - 129
1,2,3,4,7,8,9-HpCDF	50	43 - 58	$^{37}Cl_4$-2,3,7,8-TCDD	10	7.9 - 12.7
OCDF	100	63 - 159			

Table 4. Continuing Calibration Limits

Review the Injection Prep log sheet.

- Pass: Check that all samples have been spiked with 2 ng injection standard. Verify that final volume is 20 uL. Be sure that any dilutions or other comments are noted.
- Fail: Calculations of sample concentrations should reflect any deviations from normal injection prep parameters.

4.2.4 Quality Control Work Groups

The following elements should be present in a complete work group file:

- LMB topsheets
- LMB totals sheets
- LMB chromatograms (11 pages)

- OPR topsheets
- OPR chromatograms
- Extraction log sheet
- Cleanup log sheet
- ASE/Cleanup observation forms
- Dry weight sheet (where applicable)
- Any additional information (ex. re-extract request sheet)

The following procedure should be used for reviewing a work group:

- Review the header information on the LMB topsheets. Verify that the method and client sample ID (LMB or OPR) are correct.
- Review the footer information on the LMB and OPR topsheets. Verify that the following information is correct: Paradigm sample ID or OPR project number, extraction date, analysis date, method, matrix, sample weight/volume, percent solids/lipids, pH, work group number, sample datafile, retcheck datafile, beginning cal datafile and ICal datafile.
- Verify that no target analytes or EMPCs are present in the LMB above Method 23's Minimum Levels. If target analytes or EDL's are above this limit, the associated samples must have concentrations that exceed 10 times the LMB concentration for the specified analyte. Otherwise, samples must be re-extracted.
- Review the totals data for the LMB. Be sure that any ghosting peaks are removed from the totals concentrations and the associated detection limits are elevated to reflect the subtracted peaks.
- Verify that extraction and cleanup standard recoveries are within method specifications (see Table 5) for the LMB and OPR. These recoveries are found on the topsheets. Validate any failures based upon signal to noise and acceptable detection limits. If the lab validation fails a corrective action is required. Corrective actions may include re-extraction, re-cleanup, lower sample volume, extract dilution, etc.
- Verify that the recoveries in the OPR meet Paradigm's recovery limits, found in Table 6.

Compound Name	Amount Spiked (pg/µL)	Limits %
$^{13}C_{12}$-2,3,7,8-TCDD	100	25 - 164
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	25 - 181
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	32 - 141
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	28 - 130
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	23 - 140
$^{13}C_{12}$-OCDD	200	17 - 157
$^{13}C_{12}$-2,3,7,8-TCDF	100	24 - 169
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	24 - 185
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	21 - 178
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	26 - 152
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	26 - 123
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	29 - 147
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	28 - 136
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	28 - 143
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	26 - 138
$^{37}Cl_4$-2,3,7,8-TCDD	10	35 - 197

Table 5. Labeled Standard Recovery Limits

Analyte	Amount Spiked (pg/µL)	Limit (pg/µL)
2378-TCDD	10	6.7-15.8
12378-PeCDD	50	35-71
123478-HxCDD	50	35-82
123678-HxCDD	50	38-67
123789-HxCDD	50	32-81
1234678-HpCDD	50	35-70
OCDD	100	78-144
2378-TCDF	10	7.5-15.8
12378-PeCDF	50	40-67
23478-PeCDF	50	34-80
123478-HxCDF	50	36-67
123678-HxCDF	50	42-65
123789-HxCDF	50	39-65
234678-HxCDF	50	35-78
1234678-HpCDF	50	41-61
1234789-HpCDF	50	39-69
OCDF	100	63-170
^{13}C-2378-TCDD	100	20-175
^{13}C-12378-PeCDD	100	21-227
^{13}C-123478-HxCDD	100	21-193
^{13}C-123678-HxCDD	100	25-163
^{13}C-1234678-HpCDD	100	26-166
^{13}C-OCDD	200	26-397
^{13}C-2378-TCDF	100	22-152
^{13}C-12378-PeCDF	100	21-192
^{13}C-23478-PeCDF	100	13-328
^{13}C-123478-HxCDF	100	19-202
^{13}C-123678-HxCDF	100	21-159
^{13}C-123789-HxCDF	100	17-205
^{13}C-234678-HxCDF	100	22-176
^{13}C-1234678-HpCDF	100	21-158
^{13}C-1234789-HpCDF	100	20-186
^{37}Cl-2378-TCDD	10	3.1-19.1

Table 6. OPR Recovery Limits

4.3 Data Review

4.3.1 Procedure

- Complete Data Review Checklist (Section 4, Appendix A)

4.3.2 Calculations

4.3.2.1 Target compound calculation

- $$\text{PCDD/PCDF (ppt)} = \frac{\text{(Sum Ion Abun. of analyte)(ES Amount)}}{\text{(Sum Ion Abun. of Int. Std)(RRF from ICal)(Amt. of Sample)}}$$

- EMPC (ppt) = $\frac{\text{(Sum Ion Abun. of analyte)(ES Amount)}}{\text{(Sum Ion Abun. of Int. Std)(RRF from ICal)(Amt. of Sample)}}$

- EDL = $\frac{\text{2.5 (Height of Noise)(Std. Amount)}}{\text{(Height of Noise from Int. STD.)(RF from ICal)(Amt. of Sample)}}$

The instrumentation software calculates the noise level. However, manual noise determination may be employed at the reviewer's discretion in order to more accurately report peaks of interest.

4.3.2.2 Extraction Standard Recovery Calculation

- % Recovery = $\frac{\text{(Sum Ion Abun. of ES)(JS Amount)}}{\text{(Sum Ion Abun. of JS)(ES RRF from ICal)(ES Amount)}}$

The clean-up standard recoveries are calculated as above, substituting the ion abundances from the individual clean-up standard for the extraction standard

4.3.3 Requests for Re-extraction

Review all supporting data, including spike profiles, extraction logs, clean-up logs, injection prep logs, observation forms, and the sample tracking forms in the folder. The project or work group folder may contain exceptions or changes to routine spiking procedures.

Check the sample for problems relating to analysis. These problems include response factors that may introduce quantitative errors, interference that could be diluted out, or any interference that causes de-tuning or chromatographic conditions that could lead to quantitative errors.

The Laboratory Supervisor or Director should be consulted when re-extraction is considered.

If re-extraction is necessary, complete the Re-Extraction Form, which indicates the sample id, re-extraction due date, and reason for re-extraction (ref. form DC18).

When the GC/MS analyst receives the form, the samples are marked "REX" in the LIMS. The Sample ID will receive an "R" suffix. If a sample requires a second or third re-extraction, the sample id suffix will change to S, then T, and so on. The sample id with the suffix is used in all paperwork. (extraction, clean-up, injection prep, and run logs).

4.4 Reference Method

"Guidelines Establishing Test Procedures for the Analysis of Pollutants; EPA Method 1613," *Federal Register*, Vol. 62(178): 48393-48442, September 15, 1997; *Final Rule*.